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METHODS OF CHEMICAL ANALYSIS OF SILVER ANODES
(First Revision)

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Indian Standard
METHODS OF CHEMICAL ANALYSIS OF SILVER ANODES
(First Revision)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SILVER ANODES

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 March 1973, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Silver anodes used in electroplating should be of high purity and, therefore, accurate analysis of silver and copper is necessary to determine the quality of anodes. This standard was originally published in 1962. Need was felt to replace the colorimetric method for copper by a more sensitive photometric method.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes methods for determination of silver and copper in the ranges specified in IS : 1959-1961†.

2. SAMPLING FOR CHEMICAL ANALYSIS

2.1 Small pieces weighing about 10 g shall be cut preferably from opposite ends and thoroughly mixed up. Before taking samples, all surface dirt shall be removed.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960‡) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Rules for rounding off numerical values (*revised*).

†Specification for silver anodes for electroplating.

‡Specification for water, distilled quality (*revised*).

4. DETERMINATION OF SILVER

4.1 The procedure as given in **5** or **6** of IS : 2113-1972* shall be followed.

5. DETERMINATION OF COPPER BY DIETHYL DITHIOCARBAMATE (PHOTOMETRIC) METHOD (FOR 0·01 TO 0·1 PERCENT COPPER)

5.1 Outline of the Method — Silver is removed by precipitation as chloride, and copper in the filtrate is extracted as the diethyl dithiocarbamate complex in chloroform and the intensity of the colour measured at approximately 460 nm.

5.2 Reagents

5.2.1 *Dilute Nitric Acid* — 1 : 1 and 1 : 99 (*v/v*).

5.2.2 *Sodium Chloride Solution* — 20 percent. Dissolve 20 g of sodium chloride in 100 ml of water.

5.2.3 *Ammonium Hydroxide Solution* — 1 : 1 (*v/v*).

5.2.4 *Ammonium Citrate Solution* — Add 200 g of citric acid in small portions to 420 ml of dilute ammonia (1 : 1). Make the solution slightly ammoniacal, add 5 ml of sodium diethyl dithiocarbamate and extract traces of copper with chloroform, discard the chloroform extract. Dilute the aqueous phase to 500 ml.

5.2.5 *Cresol Red Indicator Solution* — Dissolve 0·1 g of the reagent in 00 ml of 50 percent rectified spirit (conforming to IS : 323-1959†).

5.2.6 *Sodium Diethyl Dithiocarbamate Solution* — 0·1 percent. Dissolve 0·1 g of the reagent in 100 ml water and keep in amber-coloured bottle.

5.2.7 *Chloroform* — conforming to IS : 5296-1969‡.

5.2.8 *Sodium Sulphate* — solid.

5.2.9 *Standard Copper Solution* — Dissolve 0·500 g of electrolytic copper in 5 ml of concentrated nitric acid, transfer to a 500-ml volumetric flask and dilute to 500 ml. Pipette out 10 ml and dilute to 1 000 ml. One millilitre of the solution contains 0·01 mg of copper (as Cu).

5.3 Procedure

5.3.1 Take 0·500 g of the sample in a 250-ml beaker and dissolve in 5 ml of dilute nitric acid (1 : 1). Dilute to 50 ml. Add 5 ml of sodium

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*Methods for assaying of silver in silver and silver alloys (*first revision*).

†Specification for rectified spirit (*revised*).

‡Specification for chloroform, technical and analytical.

chloride solution, stir, dilute to 100 ml and allow to settle. Filter through a dry Whatman filter paper No. 42. Collect the filtrate in a clean and dry 100-ml volumetric flask.

5.3.2 Transfer an aliquot containing (0·005 to 0·05 mg of copper) to a 100-ml separating funnel. Add 5 ml of ammonium citrate, and a few drops of cresol red indicator solution. Add ammonium hydroxide solution drop by drop, till the solution turns red and then 5 ml in excess. Adjust the volume to 50 ml, add 5 ml of sodium diethyl dithiocarbamate and extract with 10 ml of chloroform. To the chloroform extract, add a few crystals of anhydrous sodium sulphate to make the solution clear if necessary. Transfer the extract to a 1-cm cell and take the photometric readings at 460 nm. Run a blank using the same quantities of reagents. Correct for the blank.

5.3.3 *Calibration Curve* — Transfer 0, 0·5, 1, 2, 4 and 5 ml of standard copper solutions to different separating funnels and proceed as in **5.3.2**. Record the photometric readings and draw a calibration curve by plotting photometric readings against milligrams of copper.

5.3.4 *Calculation* — Convert the photometric readings into milligrams of copper in the aliquot taken by means of the calibration curve and calculate the percentage of copper as follows:

$$\text{Copper, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of copper in the aliquot taken, and
B = mass in g of the sample represented by the aliquot taken.

INDIAN STANDARDS

ON

PRECIOUS METALS

IS:

639-1965	Gold leaf (*revised*)
1417-1971	Grades of gold and gold alloys (*first revision*)
1418-1972	Method for assaying of gold in gold and gold alloys (*first revision*)
2112-1962	Grades of silver and silver alloys
2113-1962	Methods for assaying of silver in silver and silver alloys (*first revision*)
2270-1965	Methods for assaying of platinum and platinum alloys
2271-1967	Recommended method for spectrographic analysis of platinum
2275-1963	Grades of platinum
2278-1963	Fine gold bar, sheet, wire, granules and token (*LAGDI* or *MOHUR*)
2279-1963	Fine silver bar, sheet, wire, granules and token (*LAGDI* or *MOHUR*)
2767-1964	Gold thread (silver base)
2790-1964	14, 12 and 9 carat gold
3088-1967	Method for assaying of fine grade palladium
3095-1965	Solders for use in goldware
3096-1965	Fine grade palladium
3110-1965	Silver leaf
3111-1965	Silver thread
3112-1965	Gold and silver embroidery material
3541-1966	Code of practice for manufacture of 14 and lower carat gold alloys
3571-1966	Dental gold solders
3578-1966	Dental gold alloy wire
3610-1966	Dental gold foil
4134-1967	Recommended colour classification of rough diamond
4704-1968	Silver-tin dental amalgam alloy
4705-1968	Dental mercury
4799-1968	Dental casting gold alloys
5320-1969	Fine silver ingot
5954-1970	Dental white gold alloys
6018-1971	Platinum, platinum-rhodium catalyst gauzes
6019-1971	Platinum dish
6170-1971	Platinum crucible and lid

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